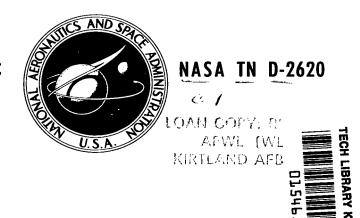
NASA TECHNICAL NOTE



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SUMMARY

A study of the effects of 1.2 and 0.30 Mev electrons on the optical-transmission properties of several transparent materials is reported herein. The materials studied were synthetic annealed sapphire, synthetic fused silica, fused quartz, and other silica glasses. The spectral range examined was 0.25 micron to 2.7 microns. Sapphire showed little sensitivity to radiation. Synthetic fused silica also showed little sensitivity except in ultraviolet transmission; however, the effects on other silica materials varied considerably. Discoloration appeared in most of the fused quartzes and other silica glasses after irradiation. Very little degradation occurred in the infrared transmission in any of the materials. It was found that heat and ultraviolet radiation have a bleaching effect on quartz damaged by radiation.

INTRODUCTION

The particulate radiation of earth radiation belts poses a serious problem to many components of satellites orbiting the earth (ref. 1). Components such as solar cells, transparent materials used as solar-cell covers, and components in optical instruments are especially sensitive to radiation.

Glasses are known to darken when exposed to radiation. There is a large quantity of data available on the effects of gamma and neutron radiation on some glasses (refs. 2 to 5), but very little data are available on electron-radiation effects (ref. 6) on the large number of commercially available silica glasses and sapphire. The present study was undertaken to provide some of these data and to assist in a proper choice of protective windows for solar cells as well as for optical instruments and manned-spacecraft windows. The optical transmission of several materials was measured before and after irradiation with 1.2 Mev and 0.3 Mev electrons. Spectral transmission tests were made for the range from 0.25 micron (μ) to 2.7 microns (μ). Transmission measurements were also made with a silicon solar cell which covered the response band from 0.40 μ to 1.20 μ (wide-band transmission). Fluorescence, visible discoloration, and bleaching of the materials were also examined.

It is known that heat and ultraviolet light have a bleaching effect on radiation-darkened glasses (refs. 2 to 4). Some bleaching tests were made to determine whether or not this effect is significant in space applications.

The materials studied were commercially supplied synthetic crystalline sapphire, synthetic fused silica, fused quartz, natural crystal quartz, non-browning lime and lead glass, high-density lead glass, Micro-Sheet, Feurex (borosilicate glass), plate glass, and Solex.

APPARATUS AND PROCEDURE

Irradiation was performed with a 1 Mev electron accelerator which is described in reference 7. The experimental setup for irradiation is shown in figure 1. The electron beam entered from the left and was scanned vertically at a rate of about 10 cps at the scan magnets. The beam then impinged upon the target in air outside the scan horn after passing through the titanium window. The electron-beam uniformity was determined to be ±5 percent over an area of 4 by 12 inches by using cobalt-glass dosimetry slides. The test samples were irradiated with 1.2 Mev and 0.30 Mev electrons. The kinetic energy of the incident electrons was determined by measuring the extreme range of electrons in aluminum and checked by scintillation-crystal measurements of the end-point bremsstrahlung energy in various materials. The dose and dose rate were monitored by an insulated 1- by 2-cm aluminum pickup located at the center of the sample tray shown in figure 2. The electron current captured by this pickup was passed to ground through an integrating electrometer. The dose rate throughout the tests was 0.03 $\mu a/cm^2$ (approx. 1.88 \times 10¹¹ e/cm² sec). The maximum dose given to most samples was 2.7×10^{15} e/cm² with 1.2 MeV electrons. This dose was estimated in September 1962 to be equivalent to approximately a 1-year dose for Explorer XVI in its orbit. The materials most commonly used as solar-cell covers (synthetic sapphire, synthetic fused silica, and Micro-Sheet) were given total doses as high as 10^{17} e/cm^2 to determine the effect of higher doses on these materials. Sapphire, fused silica, Micro-Sheet, and fused quartz were tested at 0.30 Mev for energy-comparison purposes.

The materials tested were commercially supplied synthetic crystalline sapphire, synthetic fused silica, fused quartz, natural crystal quartz, non-browning lime and lead glass, high-density lead glass, Micro-Sheet, Feurex, plate glass, and Solex. In preparation for irradiation the test samples were cut, polished, and cleaned, after which, the transmission was measured. The samples were then placed on an aluminum plate, as shown in figure 2, to be placed in the electron beam.

Preirradiation and postirradiation measurements of transmission were made by using a spectrophotometer and a wide-band transmission monitor. The monitor consisted of a tungsten light operated at a color temperature of 2800° K and an intensity of 100 mw/cm², a solar cell at room temperature, and a milliammeter to read out the short-circuit current of the cell. The wide-band transmission was obtained from the ratio of the short-circuit current of the solar cell covered with the test sample to the short-circuit current of the cell uncovered.

Although the repeatability of this method is within 0.5 percent and describes the effects on solar-cell shields, caution must be taken when applying the results to cases other than solar cells because of the low energy distribution of the lamp and the low spectral response of the cell in the visible region. The resolution of the spectrophotometer was about 10 angstroms, and measurements were repeatable within 2 percent. Fluorescence tests were made by exposing the samples to ultraviolet light and visibly noting fluorescence.

Tests were carried out to check the bleaching of the discolored materials by heat and ultraviolet light. In the heat bleaching test, samples were placed in an oven and heated at 450° F and 600° F for several hours. The ultraviolet tests were conducted by exposing the samples to the radiation from a high-intensity mercury lamp for approximately 90 hours.

RESULTS AND DISCUSSION

The materials tested were thought to be feasible for use as solar-cell covers, optical component materials, and spacecraft windows. Wherever possible, samples of thicknesses greater than, or not much less than those corresponding to the range of electrons in the material were obtained. This was possible except in the case of thin Micro-Sheet. Range-energy curves are given for electrons in aluminum (Al), silicon (Si), and silica (SiO₂) in figure 3 (from ref. 8). The thicknesses corresponding to the range R of 1.2 Mev electrons in silica (SiO₂) and sapphire (Al₂O₃) are 0.088 inch (0.494 gm/cm²) and 0.051 inch (0.517 gm/cm²), respectively. The thicknesses corresponding to the ranges for 0.30 Mev electrons in these materials are 0.026 and 0.008 inch. Values for sapphire (Al₂O₃) were obtained from the curve for aluminum. All results except those for Micro-Sheet at 1.2 Mev may be compared directly.

Results are presented for a typical sample of each type of material. Wideband transmission results are listed in the tables for comparison. Samples are identified by a manufacturer and trade name.

Sapphire

The purity of the synthetic crystalline sapphire as stated by the manufacturer was 99.99+ percent. A listing of samples and results of tests with 1.2 Mev electrons are given in table I and figure 4. Thicknesses of the samples were 0.040, 0.080, and 0.120 inch. Results indicate that a dose of $2.7 \times 10^{15} \, \mathrm{e/cm^2}$ of either 1.2 Mev or 0.30 Mev electrons does not noticeably affect the spectral or wide-band transmission of sapphire; however, a dose of $1.0 \times 10^{17} \, \mathrm{e/cm^2}$ at 1.2 Mev slightly decreased transmission in the ultraviolet region. Wide-band transmission at this dose remains unaffected because the transmission decrease occurs at wavelengths outside of the wide-band region. Close visible comparison of an irradiated and a nonirradiated sample shows that the irradiated sample darkened slightly. The fluorescence tests show that sapphire does not fluoresce before or after irradiation.

Synthetic Fused Silica

Samples of synthetic fused silica were obtained from several sources. A listing of samples and test results are given in table II and sample results are presented in figure 5. This material, which is manufactured by a vapor deposition method, may contain less than 0.2 ppm impurities. The results in table II indicate that extremely large doses of 1.2 Mev electrons are required to produce damage in wide-band transmission. Figure 5 shows the spectral transmission after several doses. Results indicate that this material degraded mainly in ultraviolet transmission when irradiated with $1.0 \times 10^{15} \ {\rm e/cm^2}$ at 1.2 MeV. This degradation extended into the visible region upon irradiation with $1.0 \times 10^{16} \ {\rm e/cm^2}$. After irradiation with $1.0 \times 10^{16} \ {\rm e/cm^2}$ the synthetic fused silica sample developed a barely detectable bluish tint. This discoloration extended approximately 0.080 to 0.090 inch into the samples which is comparable to the range of 1.2 MeV electrons in silica. Fluorescence tests showed that this material did not fluoresce before irradiation, but fluoresced red after irradiation.

The coated sample of Corning #7940 listed in table II was coated with zinc sulphide (ZnS) and magnesium fluoride (MgF) - blue reflecting and antireflecting coatings, respectively. No wide-band or spectral transmission degradation was noted. This lack of degradation is due primarily to the lack of optical transmission of the sample in the ultraviolet region where the effects of radiation damage are most prevalent in this material.

Fused Quartz

Many samples of fused quartz were tested. The samples represented more than six different trade names and were furnished by three companies. A listing of samples and test results are given in table III and sample results are presented in figures 6 to 8. This material is made by fusing natural crystal quartz, which may be as pure as 99.8 percent. Thicknesses of test samples varied from 0.0625 to 0.250 inch. The maximum dose received by most of these samples was 2.7×10^{15} e/cm² at 1.2 Mev. The Vycor sample was damaged so severely after 1.69×10^{15} e/cm² that it was not irradiated further. The results in table III show that the degree of degradation of fused quartz varied greatly from type to However, the characteristics of the damage were similar, that is, an absorption band developed as a result of irradiation with its center at approximately 0.550μ. The samples that had an Si-O absorption band at 0.240μ (fig. 7) suffered a complete distortion of this band due to the suppression of ultraviolet transmission. Associated with the absorption band at 0.550µ was a visible discoloration (deep purple). The degree of this discoloration in several samples is shown in figure 9. Some samples developed patterns that are believed to be an indication of nonuniformity of the impurity distribution and an indication of the purity of the natural quartz crystals that were fused to make the par-It is believed that impurities - mainly aluminum - are the ticular sample. cause for the discoloration (refs. 2 to 5). The depth of the darkening was also observed in the thicker samples. The depth here was approximately equal to the range of 1.2 Mev electrons in silica as was also seen in synthetic fused silica.

From fluorescence tests it was found that most of the fused quartz fluorescend red before and after irradiation; however, fluorescence was visibly indeterminate in the severely darkened samples. Irradiation of the material with 0.30 Mev electrons brought results that differ only in degree from those for 1.2 Mev electrons.

Natural Crystal Quartz

One sample of natural crystal quartz was tested. Not much is known of the origin of the particular sample; however, transmission tests showed that it was of optical quality. Results of tests are given in table IV and figure 10. An absorption band developed in the material with its center at approximately 0.46μ . This absorption band differs from the one found in fused quartz which was centered at 0.55μ . Other experimenters have found the 0.46μ band after irradiating crystal quartz with gamma radiation (ref. 5). The sample was originally water clear; however, a smoky discoloration was associated with the absorption band. There was no fluorescence before or after irradiation.

Radiation Shielding Glass

Three types of radiation shielding glass furnished by one manufacturer were tested. Results of tests made are given in table IV and figures 11 and 12. The three radiation shielding glasses tested were nonbrowning lead glass (Corning #8362), high-density lead glass (Corning #8363), and nonbrowning lime glass (Corning #8365). The nonbrowning glasses contain cerium which tends to inhibit discoloration (ref. 9). All samples were approximately 0.0625 inch thick. were irradiated with 1.2 Mev electrons. No spectral measurements were made below 0.35 µ because these materials approached a spectral cutoff in transmission in this region. Results show that there was very little damage in the Corning #8363 and Corning #8365 samples after irradiation. However, the Corning #8362 sample developed some defects and, consequently, showed appreciable damage. There was a slight darkening, and an electron discharge pattern developed (ref. 9). These patterns, known as Lichtenburg figures, were the result of a discharge of the trapped electrons through the material to an imperfection (a nick) in the otherwise smooth surface of the sample. The paths of the discharged electrons may be easily seen (fig. 13) because of the localized cracking of the glass along the paths. This Corning #8362 sample was cerium doped, and it is thought that this impurity contributes to charge storage. After charge saturation a discharge occurs. After a dose of 7.4×10^{14} e/cm² the sample was unintentionally chipped approximately 1/2 hour after removal from the beam, thus initiating the discharge.

Fluorescence tests indicated that Corning #8362 and Corning #8365 fluoresced blue before and after irradiation. Corning #8363 did not fluoresce before or after irradiation.

Micro-Sheet

Micro-Sheet is a treated, drawn, fire-polished silica glass with a low melting point. Practical thicknesses range from 0.0033 inch to 0.026 inch; the samples studied herein were 0.026 and 0.006 inch thick. This glass is often used as solar-cell covers, microscope slides, and thin film substrates. The results obtained are given in table IV and figure 14. Because of the extreme thinness of the samples, most of the incident electrons passed through the material without depositing much of their energy within the sample. Figure 14 represents a 0.026-inch-thick sample that was irradiated with $1.0 \times 10^{17} \ \rm e/cm^2$ at 1.2 Mev. Other observations showed a brownish darkening of the originally waterclear material. There was also yellow fluorescence before and after irradiation. A sample of Micro-Sheet was irradiated with 0.30 Mev electrons with damage differing only in degree from those irradiated with 1.2 Mev electrons.

Solex

Two samples of solex heat-absorbent glass were irradiated to a dose of $2.7 \times 10^{15} \ \mathrm{e/cm^2}$ at 1.2 MeV. The samples were 0.250 inch thick. Results of tests on one sample are given in table V and figure 15. This sample showed substantial radiation resistance. Only slight damage occurred in the visible region, and the discoloration was correspondingly small.

Plate Glass

A sample of soda-lime plate glass was irradiated to a dose of $1.69\times10^{15}~\rm e/cm^2$ at 1.2 Mev. Test results are given in table V and figure 16. Plate glass is very susceptible to radiation damage. A photograph of an irradiated sample of this glass is given in figure 13.

Feurex

Feurex, a borosilicate glass, was irradiated with 1.2 Mev electrons. Test results and a general description are given in table V and figure 17. This material, like plate glass, is susceptible to radiation damage. A photograph of an irradiated sample of this material is given in figure 13.

Dose Effect On Wide-Band Transmission

From wide-band transmission tests damage is plotted against total dose for irradiated materials in figure 18. Those materials that were affected severely (represented by curves c and d) degraded with a very rapid initial rate, while the lightly damaged samples (represented by curves a and b) degraded gradually.

Heat Bleaching Tests

Samples of irradiated synthetic fused silica and fused quartz were heated to test for bleaching of the radiation damage. A sample of each material was heated at 450° F and 600° F for several hours. No appreciable bleaching was found at 450° F (ref. 3). Figures 19 and 20 show the effect of heat on the optical transmission of each material. For most space considerations, heat bleaching is insignificant because the temperatures necessary for the occurrence of this phenomenon could not be tolerated by satellites in space.

Ultraviolet Bleaching Tests

Samples of fused quartz and synthetic fused silica were also exposed to ultraviolet light from a high-intensity mercury lamp for approximately 90 hours. The total ultraviolet intensity of the lamp was several times that of the sun. Substantial bleaching occurred as can be seen in figure 21 which shows an example of the results of these tests. From the ultraviolet bleaching tests, it appears that space sunlight may produce an effect on the optical transmission of a material that reduces the degradation caused by radiation. However, further work using a better simulation of solar ultraviolet light is needed to establish the actual ultraviolet bleaching rate.

CONCLUDING REMARKS

A study of the effects of 1.2 and 0.30 Mev electrons on the optical transmission properties of several transparent materials yielded the following conclusions:

- 1. Sapphire, synthetic fused silica, and some radiation-shielding glasses showed almost no damage in wide-band transmission at total doses up to $2.7 \times 10^{15} \ \mathrm{e/cm^2}$. This property makes them suitable for use as solar-cell shields. Results of spectral-transmission tests indicate these materials are also feasible for use in many optical instruments and as spacecraft windows.
- 2. The transmission of the materials tested degraded mainly in the ultraviolet and visible region.
- 3. The production of Lichtenburg figures should be considered when glasses subject to this phenomena are exposed to an electron environment, for example, cerium-doped glass.
- 4. All glasses which fluoresced before irradiation did so afterwards as far as could visually be determined. The synthetic fused silica did not fluoresce before irradiation but did afterwards.
- 5. The materials which suffered severe damage did so in the early part of exposure whereas the lightly damaged materials degraded more gradually.

6. Heat or ultraviolet light may be used to bleach discolored materials. Heat bleaching is impractical in space, but ultraviolet bleaching may be significant in reducing the degree of radiation damage. Further work is necessary to determine the full extent of this effect.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., October 10, 1964.

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TABLE I.- RESULTS OF WIDE-BAND TRANSMISSION TESTS

ON SAPPHIRE AFTER IRRADIATION

Manufacturer	Material	Thickness,	Energy, Mev	Dose, e/cm ²	Wide-band transmission loss, percent
Linde Co., Div. of Union Carbide Corp.	Linde sapphire	0.080	1.2	2.7 × 10 ¹⁵	0
		.120	1.2	1.0 × 10 ¹⁷	o
		.040	.30	2.7 × 10 ¹⁵	0

TABLE II.- RESULTS OF WIDE-BAND TRANSMISSION TESTS
ON SYNTHETIC FUSED SILICA AFTER IRRADIATION

Manufacturer	Material	Thickness,	Energy, Mev	Dose, e/cm ²	Wide-band transmission loss, percent
Engelhard Ind. (Amersil Quartz Div.)	Suprasil II	0.0625	1.2	2.7 × 10 ¹⁵	0
Corning Glass Works	Corning #7940 uv grade	0.125	1.2	2.7 × 10 ¹⁵	0
	Corning #7940 optical grade	.125	1.2	2.7 × 10 ¹⁵	o
		.250	1.2	1.0 × 10 ¹⁷	2.2
	Corning #7940 (coated)	.060	1.2	1.0 × 10 ¹⁶	0
Thermal American Fused Quartz Co.	Spectrosil A	0.125/3 pcs	1.2	2.7 × 10 ¹⁵	0
qual 62 CO.	Spectrosil B	.0637	1.2	2.7 × 10 ¹⁵	0
Dynasil Corp.	Optical grade	0.125	1.2	2.7 × 10 ¹⁵	0
		.125	1.2	1.0 × 10 ¹⁷	1.5
		.125	.30	2.7×10^{15}	0

TABLE III.- RESULTS OF WIDE-BAND TRANSMISSION TESTS
ON FUSED QUARTZ AFTER IRRADIATION

Manufacturer	Material	Thickness,	Energy, Mev	Dose, e/cm ²	Wide-band transmission loss, percent
Engelhard Ind. (Amersil Quartz Div.)	Optical grade	0.0625	1.2	2.7 × 10 ¹⁵	1.8
	Homosil	.0625	1.2	2.7 × 10 ¹⁵	2.1
	Ultrasil	.0625	1.2	2.7 × 10 ¹⁵	6.43
	Infrasil	.0625	1.2	2.7 × 10 ¹⁵	23.4
	Unknown grade	.0625	1.2	2.7×10^{15}	41.6
General Electric Co.	GE 10 ¹ 4	0.0935	1.2	2.7 × 10 ¹⁵	0.8
		.0935	.30	2.7×10^{15}	1.1
	GE 105	.0935	1.2	2.7×10^{15}	30.0
		.0935	.30	2.7×10^{15}	5.3
	GE 106	.0935	1.2	2.7 × 10 ¹⁵	28.6
		.0935	.30	2.7 × 10 ¹⁵	5.3
Corning Glass Works	Vycor (Corning #1913)	0.250	1.2	1.69×10 ¹⁵	58.9

TABLE IV. - RESULTS OF WIDE-BAND TRANSMISSION TESTS
ON SEVERAL IRRADIATED SILICA MATERIALS

Manufacturer	Material	Туре	Thickness,	Dose, 1.2 Mev, e/cm ²	Wide-band transmission loss, percent
	Natural crystal quartz	Natural crystal quartz	0.292	2.7 × 10 ¹⁵	26.8
Corning Glass Works	Corning #8362	Nonbrowning lead glass	0.0625	2.7 × 10 ¹⁵	2.4
	Corning #8363	High-density lead glass	0.0625	2.7 × 10 ¹⁵	o
3	Corning #8365	Nonbrowning lime glass	0.0625	2.7 × 10 ¹⁵	o
	Corning #0211 Micro-Sheet	Silica glass	0.006	2.7 × 10 ¹⁵	1.8
	Corning #0211 Micro-Sheet	Silica glass	0.026	2.7 × 10 ¹⁵	7.6
	Corning #0211 Micro-Sheet	Silica glass	0.026	1.0 × 10 ¹⁷	12.2

TABLE V.- RESULTS OF WIDE-BAND TRANSMISSION TESTS ON THREE COMMON GLASSES

IRRADIATED WITH 1.2 MEV ELECTRONS

Manufacturer	Material	Туре	Thickness,	Dose, 1.2 Mev, e/cm ²	Wide-band transmission loss, percent
Pittsburgh Plate Glass Co.	Solex	Heat-absorbent glass	0.250	2.7 × 10 ¹⁵	2.7
	Plate glass	Soda lime plate glass	0.250	1.69 × 10 ¹⁵	26.0
Blue Ridge Glass Corp.	Feurex	Heat-resistant borosilicate glass	0.250	2.7 × 10 ¹⁵	25.2

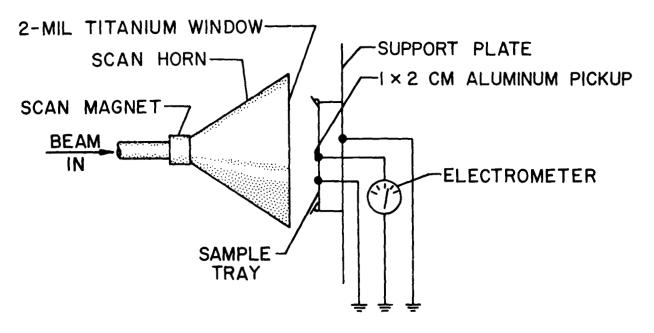
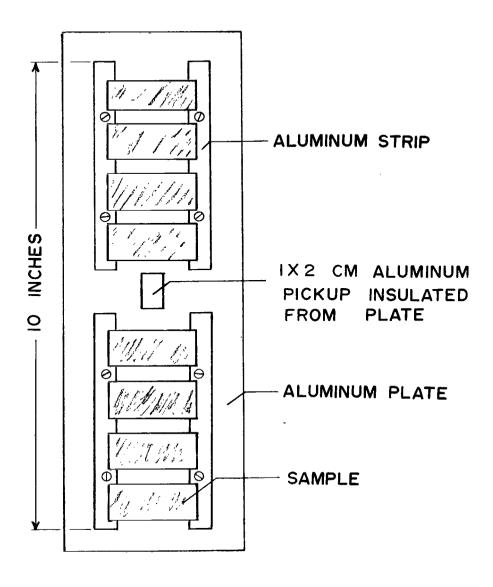


Figure 1.- Experimental setup for electron irradiation of transparent materials.



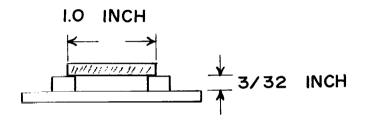


Figure 2.- Sample tray for mounting transparent materials.

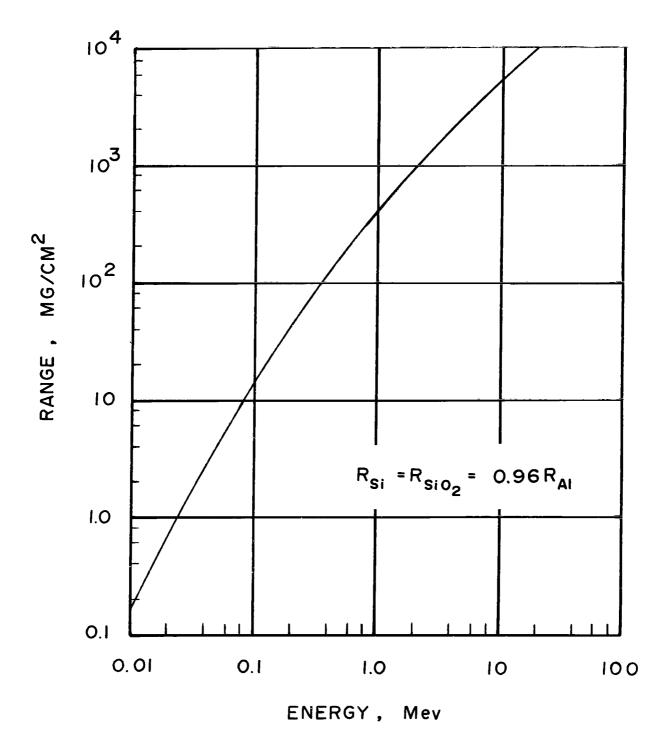


Figure 3.- Range-energy relationships for electrons in aluminum.

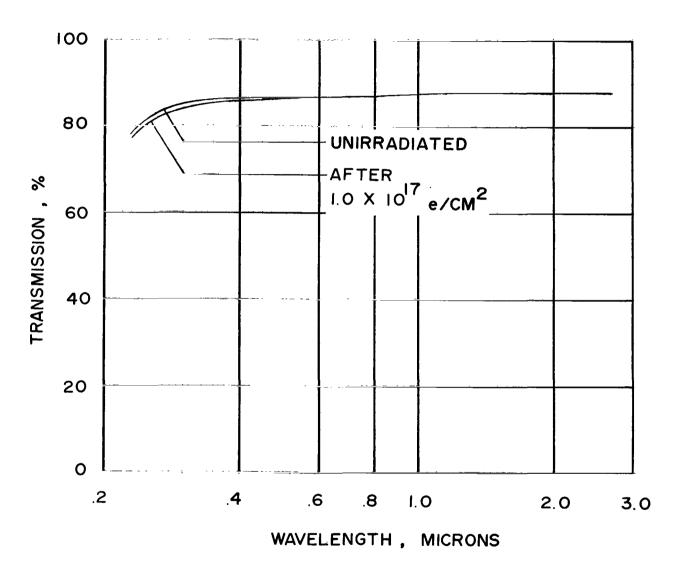


Figure 4.- Spectral transmission of synthetic annealed sapphire before and after irradiation with 1.2 MeV electrons.

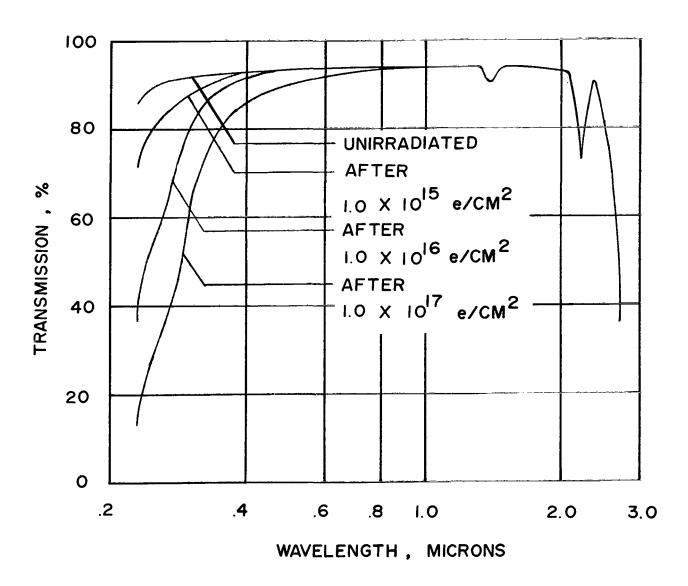


Figure 5.- Spectral transmission of a typical sample of synthetic fused silica before and after irradiation with 1.2 Mev electrons.

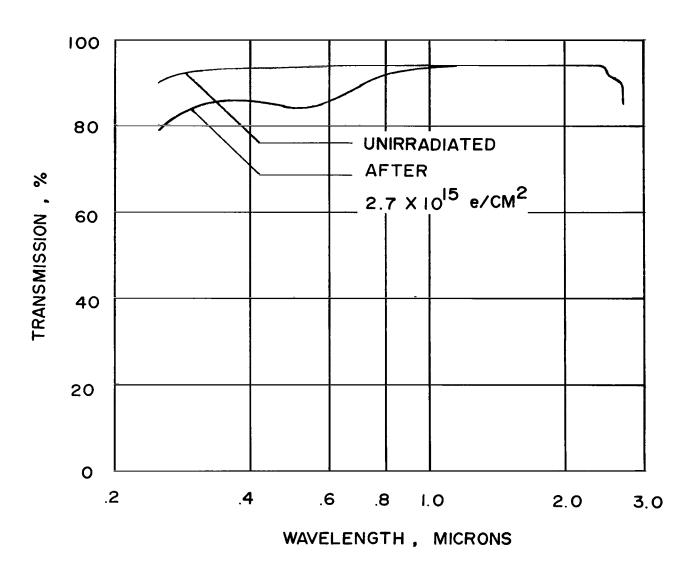


Figure 6.- Spectral transmission of Homosil fused quartz before and after irradiation with 1.2 Mev electrons.

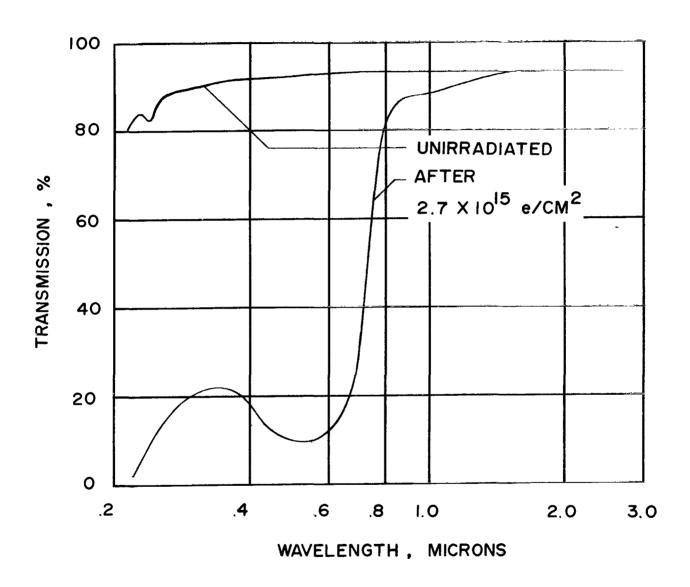


Figure 7.- Spectral transmission of GE 104 fused quartz before and after irradiation with 1.2 Mev electrons.

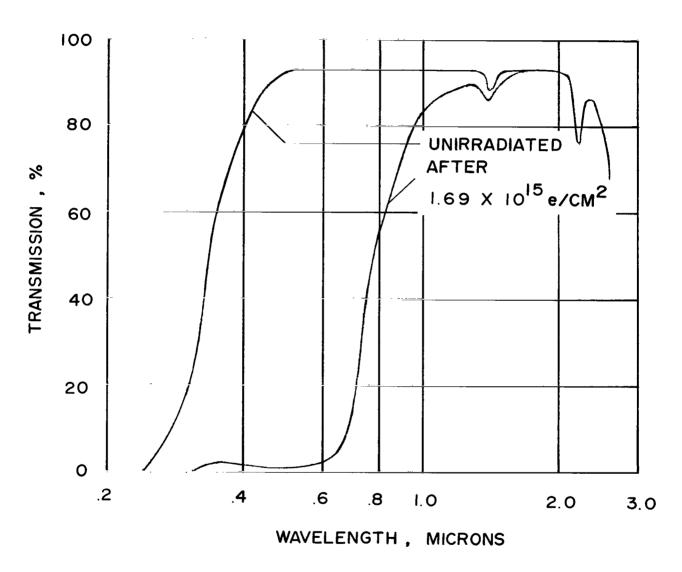


Figure 8.- Spectral transmission of Vycor before and after irradiation with 1.2 MeV electrons.

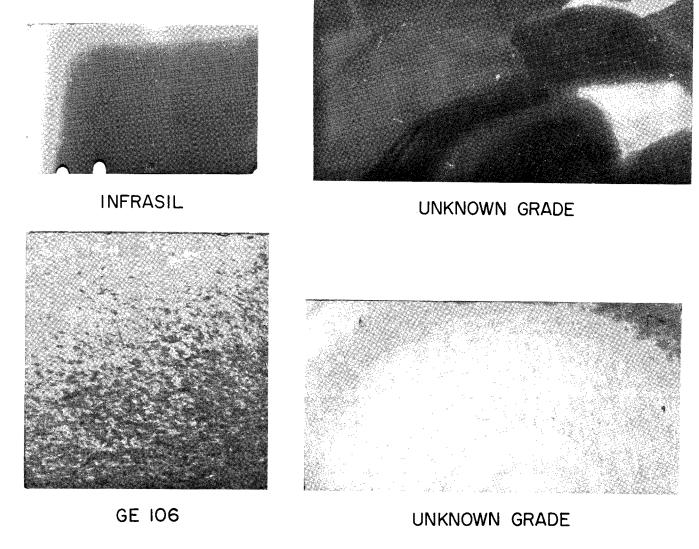


Figure 9.- Discoloration patterns of several types of fused quartz. L-64-8366

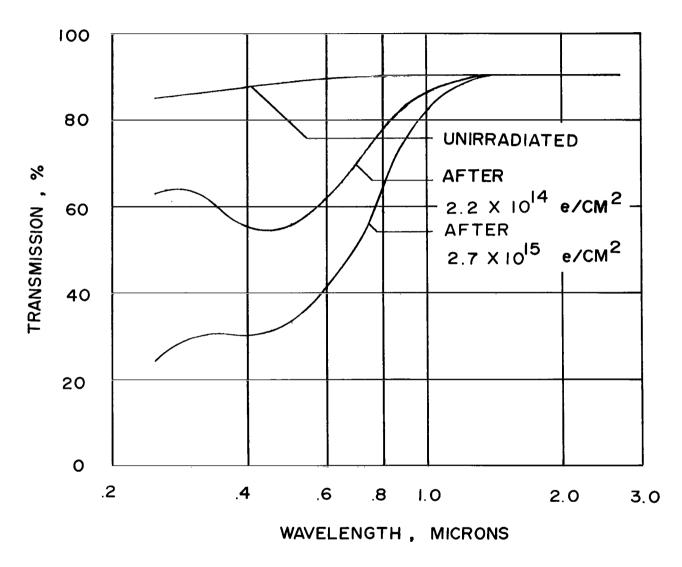


Figure 10.- Spectral transmission of natural crystal quartz before and after irradiation with 1.2 Mev electrons.

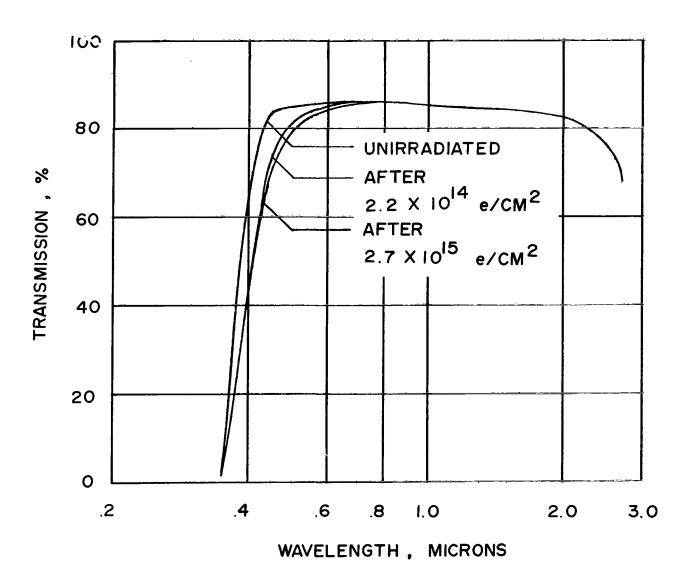


Figure 11.- Spectral transmission of Corning #8363 before and after irradiation with 1.2 MeV electrons.

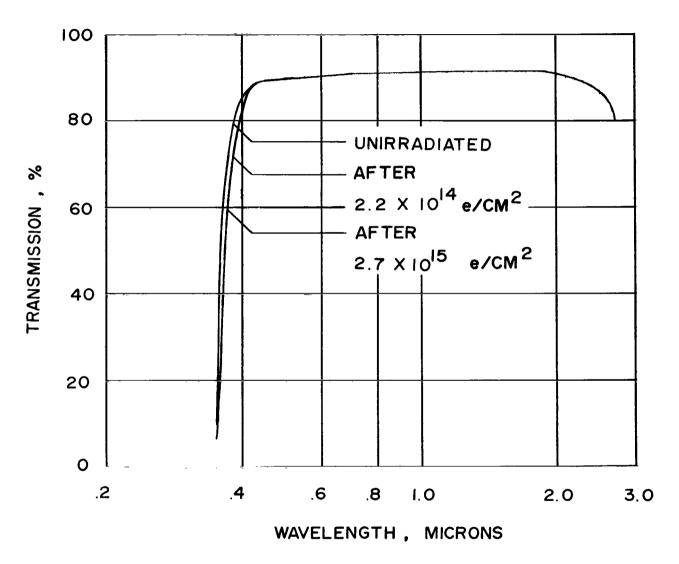
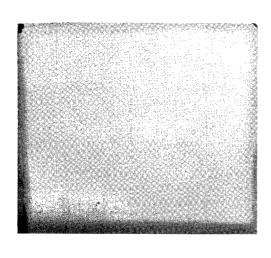


Figure 12.- Spectral transmission of Corning #8365 before and after irradiation with 1.2 Mev electrons.

CORNING #8362



FEUREX

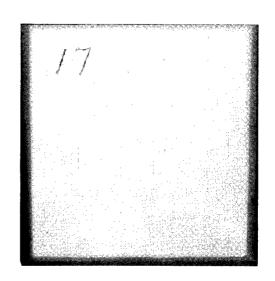


PLATE GLASS

L-64-8367

Figure 13.- Visible effect and discoloration of radiation on three types of glass.

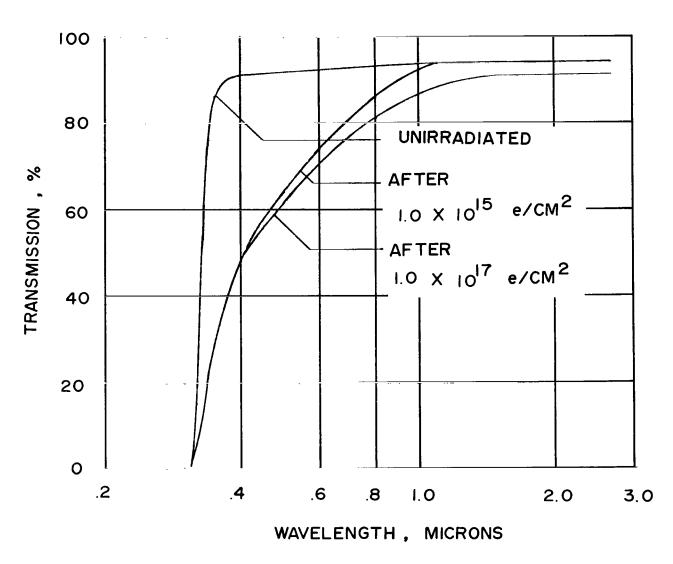


Figure 14.- Spectral transmission of Micro-Sheet before and after irradiation with 1.2 Mev electrons.

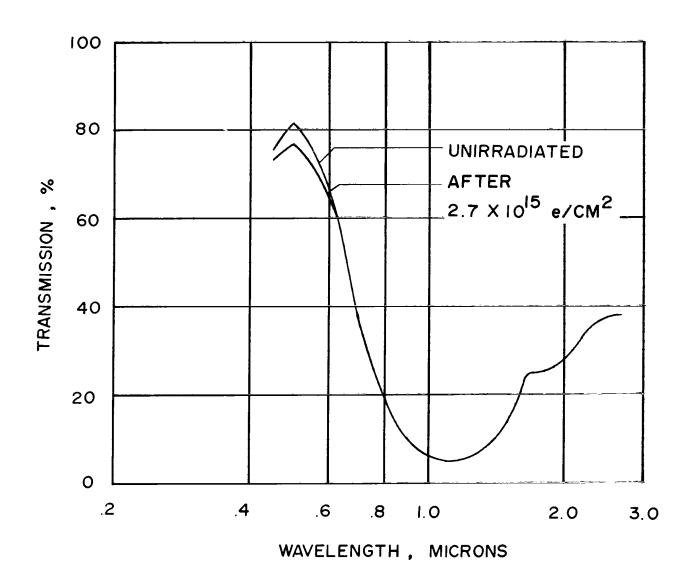


Figure 15.- Spectral transmission of Solex before and after irradiation with 1.2 Mev electrons.

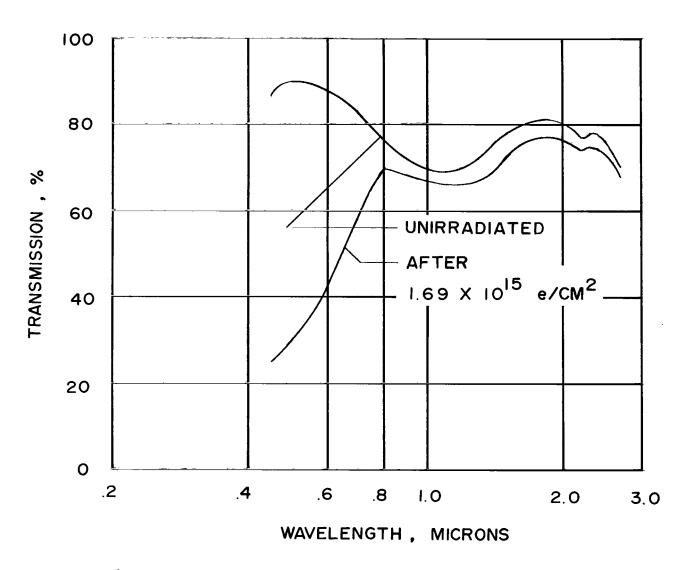


Figure 16.- Spectral transmission of soda-lime plate glass before and after irradiation with 1.2 MeV electrons.

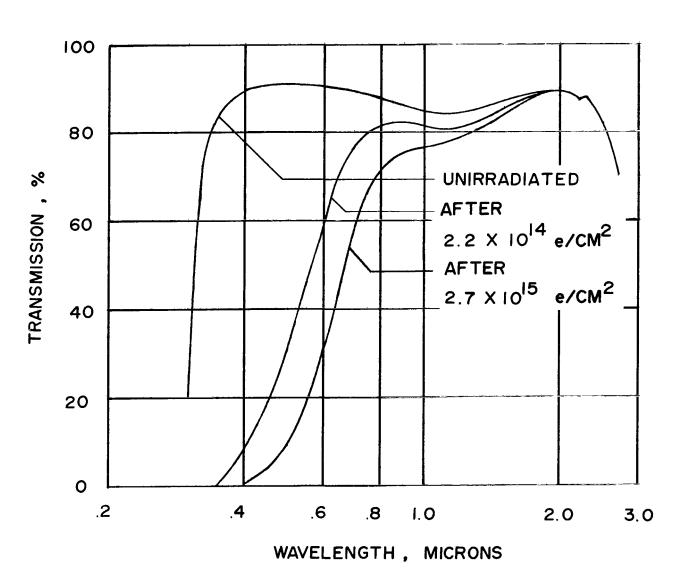


Figure 17.- Spectral transmission of Feurex before and after irradiation with 1.2 Mev electrons.

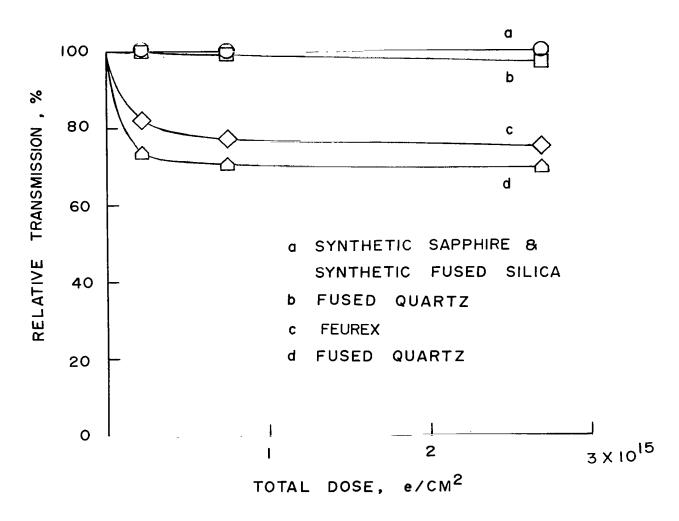


Figure 18.- Effect of total dose on wide-band transmission of several materials.

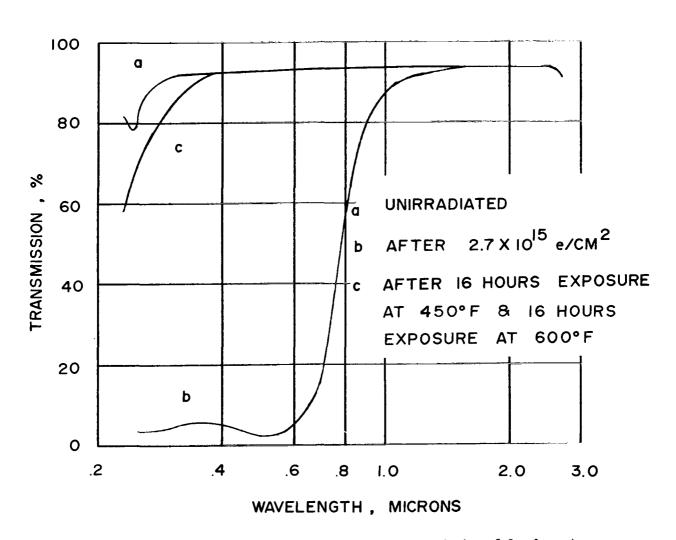


Figure 19.- Bleaching effect of heat on spectral transmission of fused quartz irradiated with 1.2 Mev electrons.

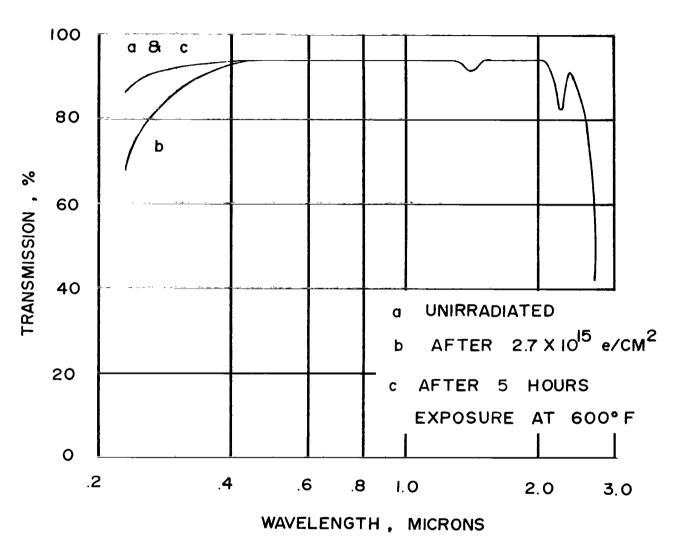


Figure 20.- Bleaching effect of heat on spectral transmission of synthetic fused silica irradiated with 1.2 Mev electrons.

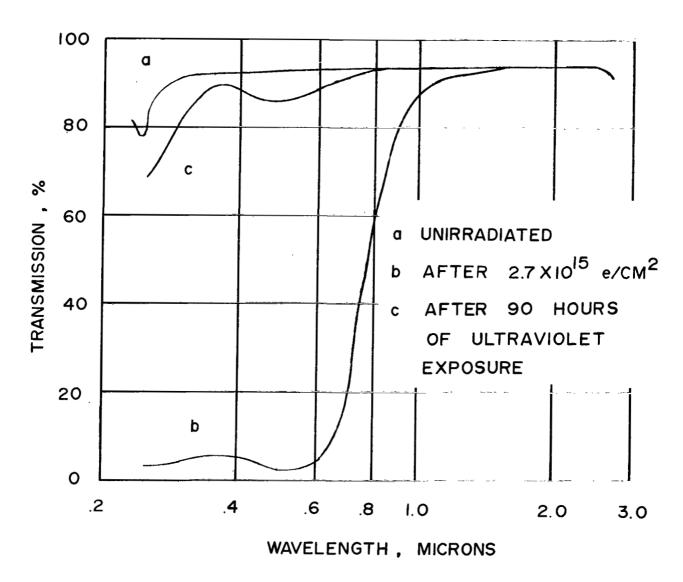


Figure 21.- Bleaching effect of ultraviolet light on spectral transmission of fused quartz irradiated with 1.2 Mev electrons.

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